UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2009/2010

November 2009

IWK 307 – Advanced Paper Technology – Instrumental Analysis of Pulp and Paper
[Teknologi Kertas Termaju – Analisa Instrumental Bagi Pulpa Dan Kertas]

Duration: 3 hours
[Masa: 3 jam]

Please check that this examination paper consists of NINE pages of printed material before you begin the examination.

[Sila pastikan bahawa kertas peperiksaan ini mengandungi SEMBILAN muka surat yang bercetak sebelum anda memulakan peperiksaan ini.]

Instructions: Answer **THREE (3)** questions. A piece of graph paper is enclosed with an answer book. Request if you need more. You may answer the question either in Bahasa Malaysia or in English.

*[**Arahan**: Jawab **TIGA (3)** soalan. Sehelai kertas graf disediakan bersama-sama buku soalan anda. Sila minta sekiranya anda memerlukan lebih daripada satu salinan. Anda dibenarkan menjawab soalan sama ada [untuk KBI] dalam Bahasa Malaysia atau Bahasa Inggeris.]*

In the event of any discrepancies, the English version shall be used.

[Sekiranya terdapat sebarang percanggahan pada soalan peperiksaan, versi Bahasa Inggeris hendaklah diguna pakai].

…2/-

1. Quantitative analysis of MOZA, which has the capability of complexing with an indicator W to form MOZA-W complex, was carried out at *ABC Paper Lab*. As an investigator, you are to take the following actions:

   (a) **Plot** the data in Table 1 on the provided graph papers to determine the amount of MOZA in three samples.

   Table 1: Absorbance for Standards and Unknowns of MOZA-W

Standards	Absorbance	Concentration
1	2	0.5 ppm
2	4	1.0 ppm
3	6	1.5 ppm
4	8	2.0 ppm
5	10	2.5 ppm
Unknown Sample	**Absorbance**	**Concentration (M)**
Sample 1	0.5	p
Sample 2	1.9	q
Sample 3	2.3	r

   NB: The unit for ppm is mg/L. The molecular weight for MOZA-W is 85 g/mole.

   (5 marks)

   (b) **Determine** the molar absorptivity, **ϵ**, of MOZA-W.

   (5 marks)

   (c) **Calculate** the value of **p, q** and **r** and state an alternative way of determining them. Are the answers derived from the two methods the same?

   (10 marks)

   (d) Ultraviolet-visible spectroscopic analysis is an analysis relying on colour intensity as response. If the colour of an indicator, W, occurred at 560 nm while the colour of MOZA-W complex occurred at 350 nm, **sketch** the ultraviolet-visible spectra of these standards. State the **assumptions** that you have to make.

   (20 marks)

…3/-

2. A hollow cathode lamp with zinc (Zn) cathode is chosen and assembled in a machine with a schematic shown as Figure 1.

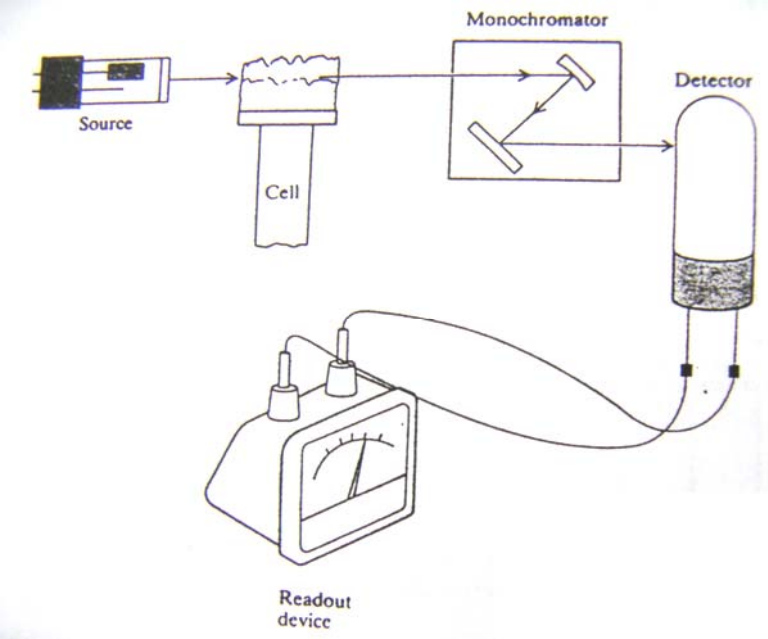


Figure 1: Schematic of an analytical instrument.

(a) **Indicate** the hollow cathode lamp (HCL) in Figure 1 by **re-sketching** it in your answer book. Indicate the cathode containing zinc and **comment** on the reusability of the lamp.

(10 marks)

(b) **Name** the instrument and **describe** the correct and successful procedure for analysis of pulp and plant biomass using this technique. **NB: Always mention the analysis that should precede this technique.**

(20 marks)

(c) You found from this analysis that the pulp contained very high amount of inorganics. If you were to move on with **further analysis**, how would you go about determining the effects of inorganics on paper **crystallinity**?

(10 marks)

3. With Appendix 1 as guide, **interpret** the hand-sketched ^{1}H-NMR spectrum of the compound in Figure 2, pointing out the necessary **corrections**. **Predict** also the mass fragmentations likely to occur.

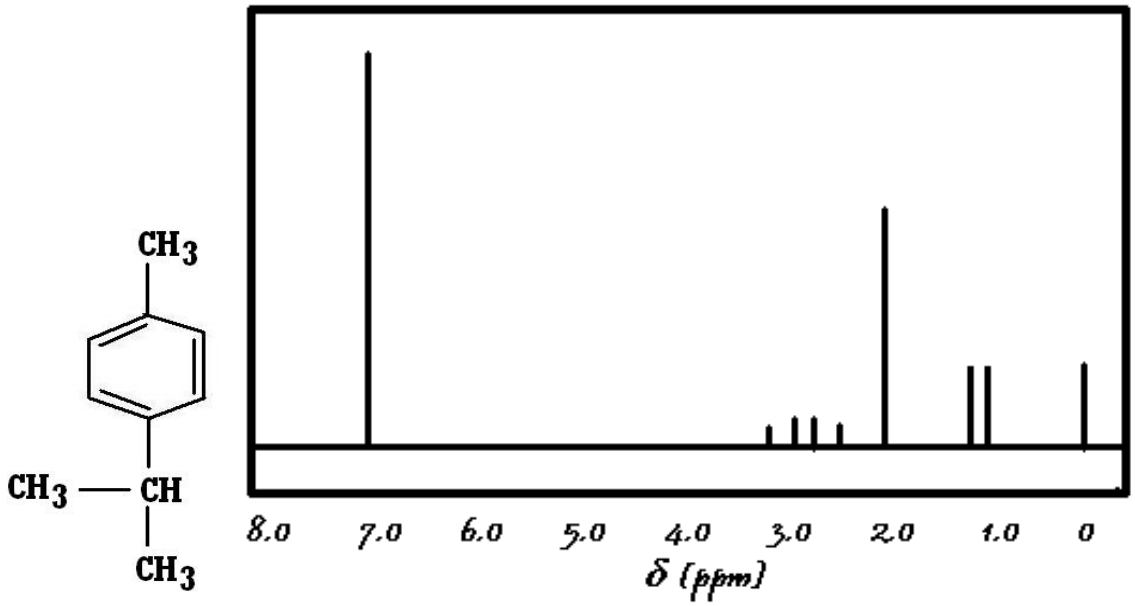


Figure 2: Compound and the corresponding ^{H}-NMR.

(20 marks)

APPENDIX 1

CHARACTERISTIC PROTON CHEMICAL SHIFTS

Type of Proton	Structure	Chemical Shift, ppm
Cyclopropane	C_3H_6	0.2
Primary	R-C**H**$_3$	0.9
Secondary	R_2-C**H**$_2$	1.3
Tertiary	R_3-C-**H**	1.5
Vinylic	C=C-**H**	4.6-5.9
Acetylenic	triple bond,CC-**H**	2-3
Aromatic	Ar-**H**	6-8.5
Benzylic	Ar-C-**H**	2.2-3
Allylic	C=C-C**H**$_3$	1.7
Fluorides	**H**-C-F	4-4.5
Chlorides	**H**-C-Cl	3-4
Bromides	**H**-C-Br	2.5-4
Iodides	**H**-C-I	2-4
Alcohols	**H**-C-OH	3.4-4
Ethers	**H**-C-OR	3.3-4
Esters	RCOO-C-**H**	3.7-4.1
Esters	**H**-C-COOR	2-2.2
Acids	**H**-C-COOH	2-2.6
Carbonyl Compounds	**H**-C-C=O	2-2.7
Aldehydic	R-(**H**-)C=O	9-10
Hydroxylic	R-C-O**H**	1-5.5
Phenolic	Ar-O**H**	4-12
Enolic	C=C-O**H**	15-17
Carboxylic	RCOO**H**	10.5-12
Amino	RN**H**$_2$	1-5

1. *Analisis kuantitatif bagi MOZA, satu bahan yang mampu menghasilkan kompleks dengan penunjuk, W, bagi menghasilkan kompleks MOZA-W telah dijalankan di sebuah makmal, ABC Paper Lab. Sebagai seorang pengkaji, anda akan mengambil langkah berikut:*

   (a) ***Plotkan*** *data dalam Jadual 1 di atas kertas graf yang disediakan bagi menentukan kandungan MOZA dalam tiga sampel.*

Jadual 1: Serapan bagi Larutan Piawai dan Sampel MOZA-W

Larutan piawai	***Serapan***	***Kepekatan***
1	***2***	***0.5 ppm***
2	***4***	***1.0 ppm***
3	***6***	***1.5 ppm***
4	***8***	***2.0 ppm***
5	***10***	***2.5 ppm***
Sampel	***Serapan***	***Kepekatan (M)***
Sampel 1	***0.5***	***p***
Sampel 2	***1.9***	***q***
Sampel 3	***2.3***	***r***

NOTA: Unit bagi ppm ialah mg/L. Jisim molekul MOZA ialah 85 g/mol.

(5 markah)

(b) ***Tentukan*** *molar absorptivity, **ϵ**, bagi MOZA-W.*

(5 markah)

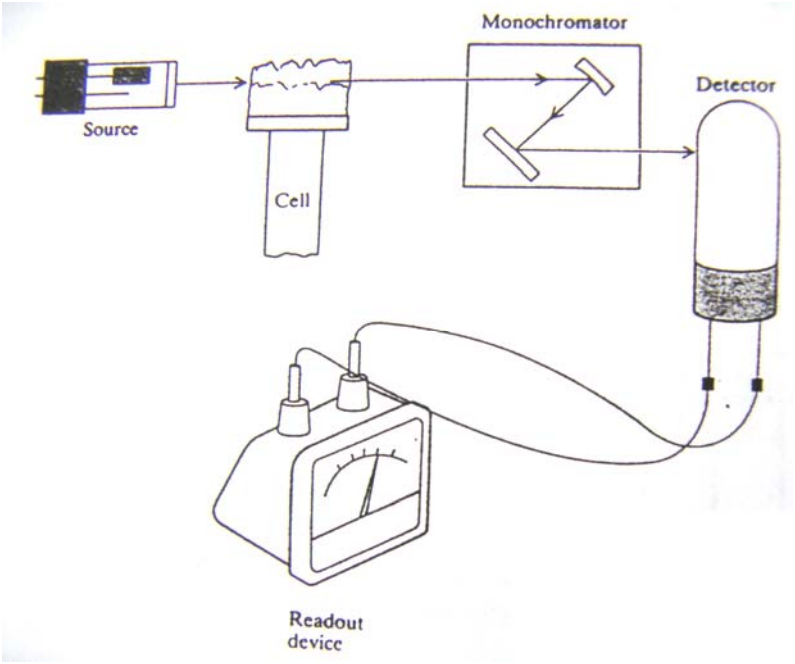
(c) ***Tentukan p**, **q** dan **r** pada Jadual 1 dan nyatakan kaedah alternatif bagi penentuan p, q dan r. Adakah kedua-dua kaedah ini memberikan jawapan yang sama?*

(10 markah)

(d) *Analisis spektroskopi ultralembayung-nampak merupakan kaedah analisis yang bergantung kepada keamatan warna sebagai respon. Sekiranya warna penunjuk W memberikan respon pada 560 nm sementara kompleks MOZA-W memberikan warna pada 350 nm,* ***lakarkan spektrum*** *bagi larutan piawai yang dianalisis. Nyatakan* ***anggapan*** *anda.*

(20 markah)

2. *Lampu hollow cathode dengan katod zink telah dipilih dan dipasang pada sebuah alat dengan skematik yang ditunjukkan pada Rajah 1.*



Rajah 1: Skematik bagi sejenis peralatan analisis.

(a) ***Tunjukkan*** *lampu hollow cathode (HCL) pada Rajah 1 melalui* ***lakaran*** *semula dalam kertas jawapan anda dan tunjukkan bahagian katod yang mengandungi zink. Berikan* ***komen*** *anda tentang keboleh-gunaan semula lampu ini.*

(10 markah)

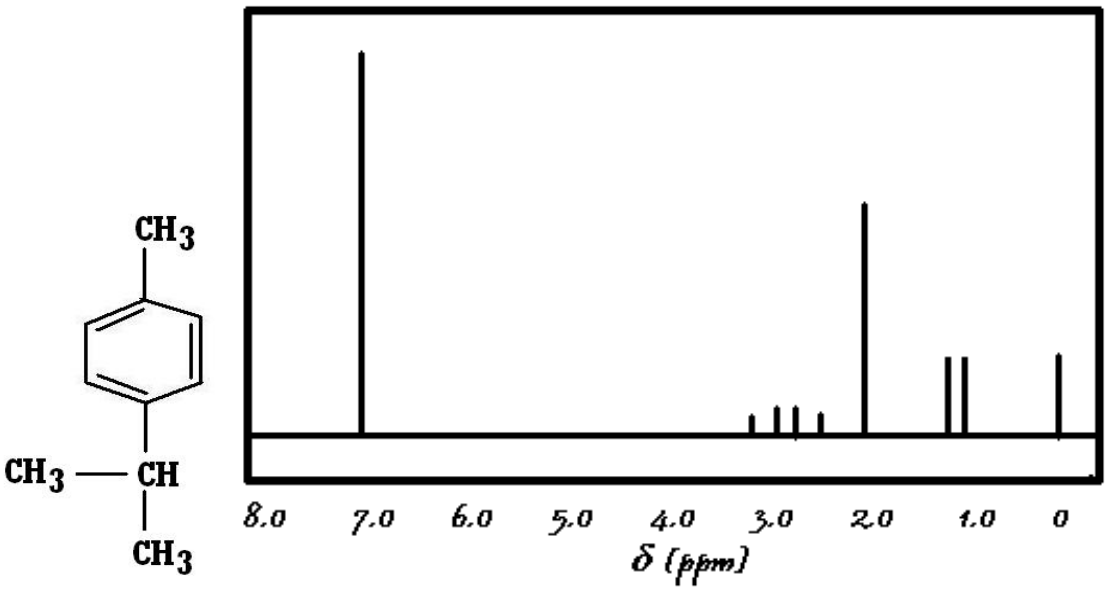
(b) ***Namakan*** *alat dalam skematik pada Rajah 1 dan* ***huraikan*** *prosidur analisis pulpa dan biojisim tumbuhan yang tepat menggunakan teknik ini.*
NOTA: Sebutkan analisis yang perlu mendahului analisis ini.

(20 markah)

(c) *Daripada analisis ini, anda mendapati bahawa pulpa yang diuji mengandungi bahan bukan organik yang amat tinggi. Jika anda perlu mengadakan **analisis lanjut** bagi memeriksa kesan bahan tersebut terhadap **kekristalan** kertas, apakah langkah yang akan anda ambil selanjutnya?*

(10 markah)

3. *Dengan merujuk kepada Lampiran 1, berikan **interpretasi** bagi lakaran ^{1}H-NMR bagi sebatian pada Rajah 2 sambil mencadangkan **pembetulan** yang perlu. **Ramalkan** juga fragmentasi jisim yang mungkin berlaku.*



Rajah 2: Sebatian dan spektrum lakaran ^{1}H-NMR.

(20 markah)

LAMPIRAN 1

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Vinylic	*C=C-H*	*4.6-5.9*
Acetylenic	*triple bond,CC-H*	*2-3*
Aromatic	*Ar-H*	*6-8.5*
Benzylic	*Ar-C-H*	*2.2-3*
Allylic	$C\text{=}C\text{-}CH_3$	*1.7*
Fluorides	*H-C-F*	*4-4.5*
Chlorides	*H-C-Cl*	*3-4*
Bromides	*H-C-Br*	*2.5-4*
Iodides	*H-C-I*	*2-4*
Alcohols	*H-C-OH*	*3.4-4*
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Aldehydic	*R-(H-)C=O*	*9-10*
Hydroxylic	*R-C-OH*	*1-5.5*
Phenolic	*Ar-OH*	*4-12*
Enolic	*C=C-OH*	*15-17*
Carboxylic	*RCOOH*	*10.5-12*
Amino	RNH_2	*1-5*